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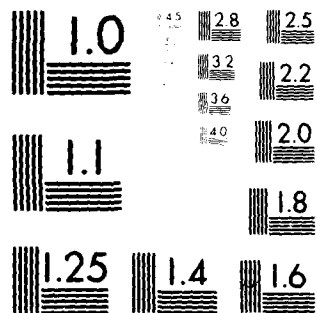
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**AN INVESTIGATION OF A COMPOSITE OXIDE FILM
AS A CORROSION INHIBITOR FOR 2024 ALUMINUM**

NEIL T. MCDEVITT

*MECHANICS AND SURFACE INTERACTIONS BRANCH
NONMETALLIC MATERIALS DIVISION*

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MORRIS BROWN UNIVERSITY*

JUNE 1980

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TECHNICAL REPORT AFWAL-TR-80-4065
Interim Report for period January 1979 — December 1979

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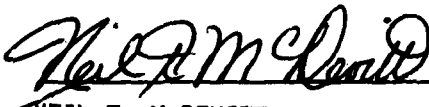
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FOREWORD

This technical report was prepared by Neil T. McDevitt, Mechanics and Surface Interactions Branch, Nonmetallic Materials Division, Air Force Materials Laboratory (AFML/MLBM), and Dr. Frank N. Weaver, Chemistry Department, Morris Brown University. This work was initiated under Project 2419 "Nonmetallic and Composite Materials:", and W.U.D. No. 44, "Improved Materials, Processes, and Life Prediction Methodology of Adhesive Bonding" monitored by T. W. Haas.

This report covers work conducted inhouse during the period January 1979-December 1979.

The authors are especially grateful to Mr. James Solomon for his helpful technical discussions and Mr. William Baun for his valuable advice. Also to Mr. Gary Jones and Mr. Douglas Hanlin for their technical assistance.

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TABLE OF CONTENTS

SECTION		PAGE
I	BACKGROUND	1
II	INTRODUCTION	2
III	EXPERIMENTAL	3
IV	RESULTS AND DISCUSSION	5
	1. CHARACTERIZATION OF OXIDE FILMS	5
	A. Phosphoric Acid Anodic Oxide	
	B. Chromate Conversion Oxide	5
	2. CHROMATE-ANODIC OXIDE COMPOSITE FILM	6
	3. SALT SOLUTION ENVIRONMENT	8
	REFERENCES	12

LIST OF ILLUSTRATIONS

FIGURE		PAGE
1	SEM Micrograph of a 1.0M H_3PO_4 , 10V, 12 Minute Anodization of Bare 2024-T3 Aluminum	14
2	Auger Depth Profile of Specimen Examined in Figure 1.	15
3	SEM Micrograph of Specimen A-044.	16
4	SEM Micrograph of Specimen A-121.	17
5	Auger Spectrum of Surface of Specimen A-121.	18
6	Auger Depth Profile of Specimen A-121.	19
7	SEM Micrograph of Specimen AP-121.	20
8	Auger Spectrum of Surface of Specimen AP-121.	21
9	Auger Spectrum of Metal-Oxide Interface of Specimen AP-121.	22
10	Auger Spectrum of Surface of Specimen AP-095.	23
11	SEM Micrograph of Specimen P-036, a) 100X, b) 500X.	24
12	Auger Spectrum of Pit and Edge of Pit of Specimen P-036.	25
13	SEM Micrograph of Specimen A-036, a) 100X, b) 500X.	26
14	Auger Depth Profile Through Center of Healed Pit Specimen on A-036.	27
15	3X Photo of Specimen AP-040 Showing the Leaching Effect of Salt Solution.	28
16	3X Photo of Specimen AP-036, X) Sulfuric-Chromic Pretreatment, Y) Nitric-Hydrofluoric Pretreatment.	29
17.	3X Photo of Specimen AP-190, X) Sulfuric-Chromic Pretreatment, Y) Nitric-Hydrofluoric Pretreatment.	30

LIST OF TABLES

TABLE		PAGE
I	Experimental Conditions for Specimens Studied.	31

SUMMARY

Placing a corrosion retardant directly in the oxide film should greatly improve the corrosion resistance of that film in comparison to the primer-inhibitor method. This study shows a retardant can be placed in the oxide film of 2024 aluminum by forming a composite oxide from the sequential generation of a chromate conversion coating-anodization process. A composite oxide film formed from a two minute conversion coating, and a 10 volt, 8 minute anodization in 1.0M H_3PO_4 showed a corrosion resistance on the average of twice that of a 10 volt, 10 minute 1.0M H_3PO_4 anodic oxide.

A follow on study has been formulated to determine if the composite oxide film maintains the bonding characteristics of the original phosphoric acid anodic oxide.

SECTION I

BACKGROUND

Extensive research programs have shown phosphoric acid anodization to be one of the better methods for preparing the surface of bare aluminum alloys for adhesive bonding.⁽¹⁾ While acting as a surface preparation in forming good metal-to-metal adhesive bonds the formed anodic oxide should also have reasonable passivation properties for the bulk metal. The passivation properties of an oxide formed in phosphoric acid are known to be minimal when compared to other anodic oxides. Consequently, the chemical breakdown properties of this oxide film are of great interest for bond-line corrosion resistance.

Corrosion control of airframe structures exposed to the environment has always been a concern⁽²⁾ of aircraft manufacturers. However, at the present time bond-line corrosion, as a primary cause of failure, does not seem to be a major concern. Since the anodic oxide offers minimal corrosion protection, and the airframe aluminum alloys contain copper, bond-line corrosion may limit the desired durability of adhesively bonded structures. A recent study⁽³⁾ on unbonded specimens has shown copper to be present in a large majority of the corrosion pits formed on the bulk aluminum alloy. A survey study to improve the corrosion resistance of the anodic oxide was recently completed.⁽⁴⁾

SECTION II

INTRODUCTION

Current technology for improving the corrosion resistance of the oxide layer incorporates corrosion inhibitors directly into the primer layer. The primer layer is placed on top of the prepared oxide layer. These inhibitors remain as particulate matter in the primer and actually lay on the surface of the oxide. In order for these inhibitors to be effective as corrosion retardants for the oxide-primer, or metal-oxide interface they must come into contact with the environment, partially dissolve, and then be physically adsorbed by these interfaces. Placing the corrosion retardant directly into the oxide film should greatly improve the corrosion resistance of that layer when compared to the primer-inhibitor method.

The initial aim of our present research program is to improve the corrosion resistance of the phosphoric acid anodic oxide film. In the preliminary survey study⁽⁴⁾ one method seemed to show some promise as a corrosion retardant for the anodic oxide film formed on 2024 aluminum. This method generated a composite oxide on the 2024 aluminum by the sequential processes of treating the deoxidized metal surface with a chromate conversion solution followed by phosphoric acid anodization.

The purpose of this report is to chemically characterize a large number of 2024 aluminum surfaces having a chromate-anodic composite oxide film and evaluate the corrosion resistance of this film with reference to a salt solution environment.

SECTION III

EXPERIMENTAL

Rectangular specimens were cut from a sheet of 2024-T3 bare aluminum alloy. Each specimen was 5.0 x 2.5 x 0.05 centimeters. All of the specimens were degreased with an acetone wipe and then ultrasonically cleaned in carbon tetrachloride for five minutes. Specimens were deoxidized with either a nitric-hydrofluoric acid (3 minutes at room temperature), or a sulfuric-chromic acid (15 minutes at room temperature) solution.

The composite oxide film was generated by a chromate conversion solution (Alodine 1201*) followed by anodization in a 1.0M phosphoric acid electrolyte. A number of different parameters were used in forming the composite oxide film and the specimens reported in Table I represent a cross section of the surfaces studied in this report.

The instruments used in this study were the ISI 60 Scanning Electron Microscope (SEM) and the PHI Scanning Auger Microprobe (SAM).

Corrosion resistance of each composite oxide film was determined by a salt solution dip. Fifteen specimens were attached to a 15-centimeter diameter wheel that was driven by a stepping motor. Each specimen was insulated in order to prevent galvanic interaction with the wheel. The specimens were

*Amchem Products, Inc.

dipped through the salt solution for one minute followed by a three minute air-dry cycle.

SECTION IV

RESULTS AND DISCUSSION

1. CHARACTERIZATION OF OXIDE FILMS

A. Phosphoric Acid Anodic Oxide

The surfaces generated by phosphoric acid on aluminum, clad aluminum alloys, and bare aluminum alloys was reported in a recent study.⁽⁵⁾ An SEM micrograph of a phosphoric acid anodized surface of bare 2024 aluminum is shown in Figure 1. An Auger depth profile of this oxide film (Figure 2) shows copper present at the metal-oxide interface.

B. Chromate Conversion Oxide

Chromate conversion oxide films are dependable corrosion resistant films when formed on aluminum alloys. They are generally applied by immersion processes. Most conversion coating solution compositions are proprietary with each manufacturer and the specific chemical constituents are very seldom known. The Alodine 1201 solution used in this study meets the requirements of Military Specification MIL-C-5541B and contains ferricyanide salts, acidic chromates, and fluorides. The immersion process dictates open circuit potential conditions. Under these conditions the surface morphology generated can usually be described as "dried mud" (Figure 3). Similar passivating oxide coatings have been studied by a number of researchers.⁽⁶⁻¹¹⁾ The thickness of the film was obtained by bending the specimen into a "U" shape and observing the cracked oxide at the apex with the electron microscope. Figure 4 shows a micrograph obtained

in this manner. The measured thickness of this film is approximately 0.4 μm . The films prepared in this study grew rapidly in the first 90 seconds, then the rate decreased until a thickness of 0.4 to 0.55 μm was obtained after four minutes. These same thicknesses were generated with eight minute immersions. Similar results were reported by Katzman.⁽⁶⁾ A SAM spectrum of the surface (specimen A-121) is shown in Figure 5. These data indicate the first part of the surface is composed of chromium and iron compounds. No aluminum compounds are detected. According to Koudelkova,⁽⁹⁾ the chromium species are Cr_2O_3 (Cr^{3+}) and CrO_4^{2-} (Cr^{6+}). An Auger depth profile (Figure 6) of this specimen shows chromium to be prevalent throughout the oxide coating. Copper appears to be present at the metal-oxide interface similar to that found in the depth profile of the anodic oxide film (Figure 2). Several specimens were made the anode in an electrolytic bath of Alodine 1201 and subjected to an applied potential of 30 volts. A SAM analysis of this specimen did not show any chromium present in the oxide layer.⁽¹²⁾ This indicates that an open circuit potential is required to place the chromium throughout the oxide film.

2. CHROMATE-ANODIC OXIDE COMPOSITE FILM

A large number (200) of specimens were prepared and examined during this study. The specimens notated in Table I are representative of surface parameters evaluated. The chromate conversion coating was grown on a deoxidized surface of bare 2024 aluminum and then dried. The specimen was then anodized in a

phosphoric acid electrolyte. Composite aluminum oxide films have been studied by Alwitt;⁽¹³⁻¹⁴⁾ however, these films were formed from the growth of a hydrated oxide and a barrier layer forming electrolyte.

Phosphoric acid is an aggressive, pore-forming electrolyte and we expected the chromate conversion coating to be thoroughly mixed with the anodic oxide. The initial SEM study on this composite oxide film showed the anodic oxide growing under the conversion coating (Figure 7). The thickness of the initial conversion coating was 0.4 μm . The anodization process thinned the conversion coating to about 0.2 μm while the anodic oxide thickness is measured at 0.26 μm . The Auger analysis of the surface of this specimen is shown in Figure 8. Chromium and iron are detected similar to the conversion coating spectrum shown in Figure 5; however, the anodization process does leave aluminum in the surface oxide that was not observed in the conversion coating. With the anodic oxide growing under the conversion coating we were interested as to the elemental condition of the metal-oxide interface. An Auger depth profile was performed on the specimen shown in Figure 7. An Auger spectrum was obtained at a point where the sputter profile indicated the oxygen peak-to-peak intensity was small (Figure 9). From all indications (Al 67eV, Cu 940eV) the Auger data was obtained close to the metal-oxide interface, with the interesting aspect that chromium (530 eV) was still detected. It is apparent, under the conditions of this anodization, that some mixing of the conversion coating and anodic oxide does take place at the metal-oxide interface.

In comparison to the above study a composite oxide film was formed at a lower voltage but four times the anodization time. The SEM micrograph showed only a singular film and the Auger spectrum (Figure 10) indicated only the presence of aluminum oxide. An Auger depth profile showed chromium to be absent throughout the thickness of the oxide film. From the work of Mackintosh⁽¹⁵⁾, it is apparent that the chromium ion is highly mobile under an applied potential. In order to keep some chromium at the metal-oxide interface the length of time of anodization had to be smaller. Composite oxide films formed at 10V and 10 minutes or less show similar Auger data as observed in Figure 9 with a small amount of chromium remaining at the metal-oxide interface.

3. SALT SOLUTION ENVIRONMENT

The most difficult aspect of any corrosion study is the method of assessment chosen to determine the breakdown of the surface under the influence of an environment. Pitting attack on the aluminum surface was the primary cause of corrosion in this study. Most specimens were masked leaving one square centimeter of surface exposed to the corrosive environment. All composite oxide specimens were run as duplicates with three phosphoric anodic oxide surfaces as controls. The corroded specimens were classified according to the size and number of pits in the one square centimeter area. The classifications were based on a purely visual and subjective assessment of appearance analogous to the charts reported by Carter.⁽¹⁶⁾

When an individual specimen was assessed as having a "moderate" number of pits present on the exposed surface it was removed from the test and the time-in test notated. Each test was composed of 15 specimens on the dipping wheel. The time-in test was used only for comparison of the duplicate specimens in the same test. It did not appear to be a valid number from one test to another. The wetting, air-dry cycle of the test produced an accelerated corrosion when compared to wet-environment-only testing and may have introduced some vagaries that would not allow for time comparison between individual tests.

Some corrosion studies were performed on the neat phosphoric anodic oxide surface and the neat chromate conversion coating. Figure 11 shows the results of a phosphoric anodic oxide surface after exposure of 15 hours to a 4 percent salt solution. Pitting was allowed past the moderate classification in this particular test. An Auger spectrum was obtained from one of the pits on this specimen (Figure 12). Data obtained from the edge of the pit indicates the presence of sodium and chlorine which is understandable. However, data obtained from the center of the pit shows only a small amount of sodium and chlorine with a larger amount of copper. The driving force of this local cell builds salt mounds at the edge of the pits while exposing the copper at the base of the pit. The chromate conversion coating required 45 hours in test (Figure 13) in order to achieve a small number of pits. In this case a true pit does not form but more like a "plugged" pit. An Auger depth profile was performed on the

center of one of these pits and a spectrum recorded after 0, 20, and 80 minutes of profiling. The zero minute scan shows only aluminum and oxygen present. After 20 minutes chromium appears to be part of the oxide layer. After sputtering completely through the oxide layers (80 minutes) we again observe the presence of copper. These data indicate a pit is caused by the environment and the chromium compounds in the oxide layer heal the pit with the formation of aluminum oxide on the external portion of the pit. Since the pit does form initially the conversion coating appears to be a retardant to the rate of corrosion. This effect can be seen in Figure 14. The coloration of the composite oxide specimens is a light gold. As the specimen goes through a wet, air-dry cycle in a clockwise direction, the gold color starts to leach out at the bottom of the specimen and eventually takes on the pattern shown in Figure 15 as the salt film is drained off the surface by the motion of the wheel.

One set of results from this study that was not expected was the role the pretreatment deoxidation step plays in the apparent overall corrosion rate. This can be seen in the photos of Figures 16 and 17 taken at 3 times normal size. The x designated photos in each figure were pretreated with the sulfuric-chromic acid solution, while the y designated photos were pretreated with a nitric-hydrofluoric acid solution. The number of pits per square centimeter were always greater for the same time period in the nitric-hydrofluoric acid pretreated surfaces when the composite oxide film formation was the same for both pretreatments. This trend was prevalent throughout this particular study.

In general the overall results from this program show the composite oxide to resist pitting corrosion twice as long as the neat phosphoric anodic oxide. However, the neat chromate conversion coating provides three times as much protection as the phosphoric anodic oxide.

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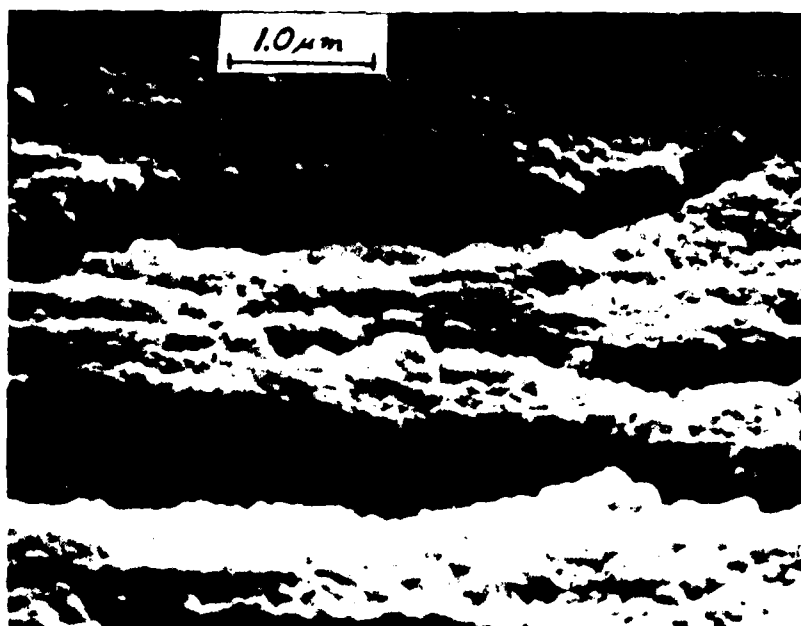


Figure 1. SEM Micrograph of a 1.0M H_3PO_4 , 10 V, 12 Minute Anodization of Bare 2024-T3 Aluminum.

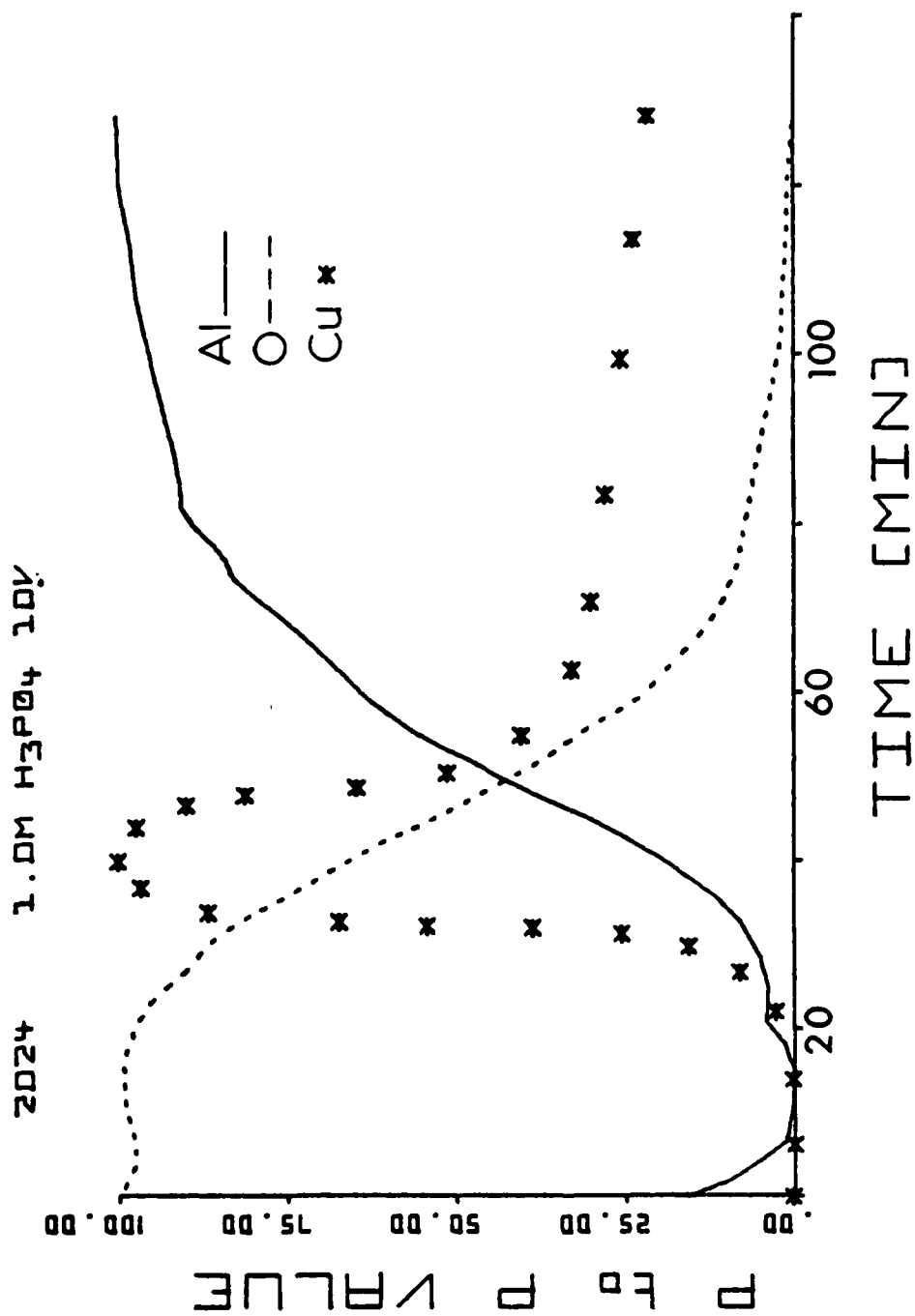


Figure 2. Auger Depth Profile of Specimen Examined in Figure 1.



Figure 3. SEM Micrograph of Specimen A-044.

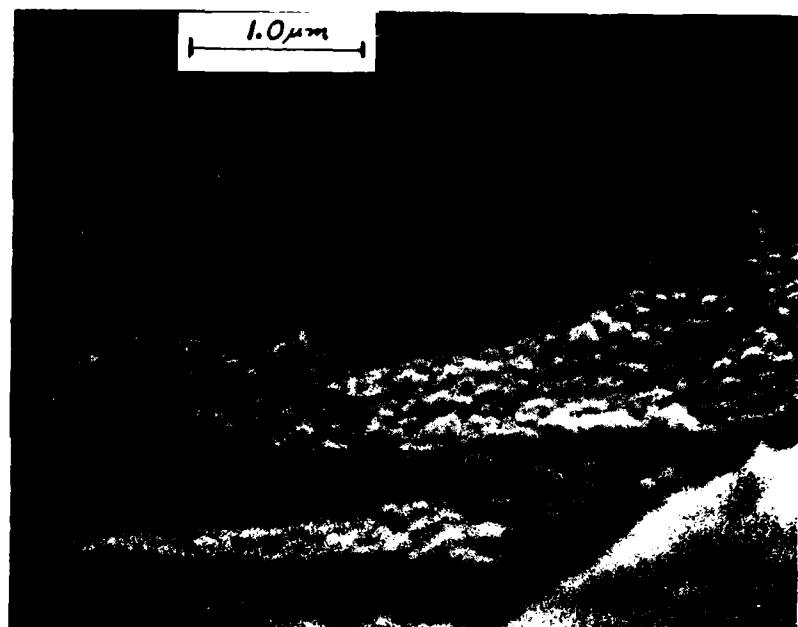


Figure 4. SEM Micrograph for Thickness
Measurement of Specimen A-121.

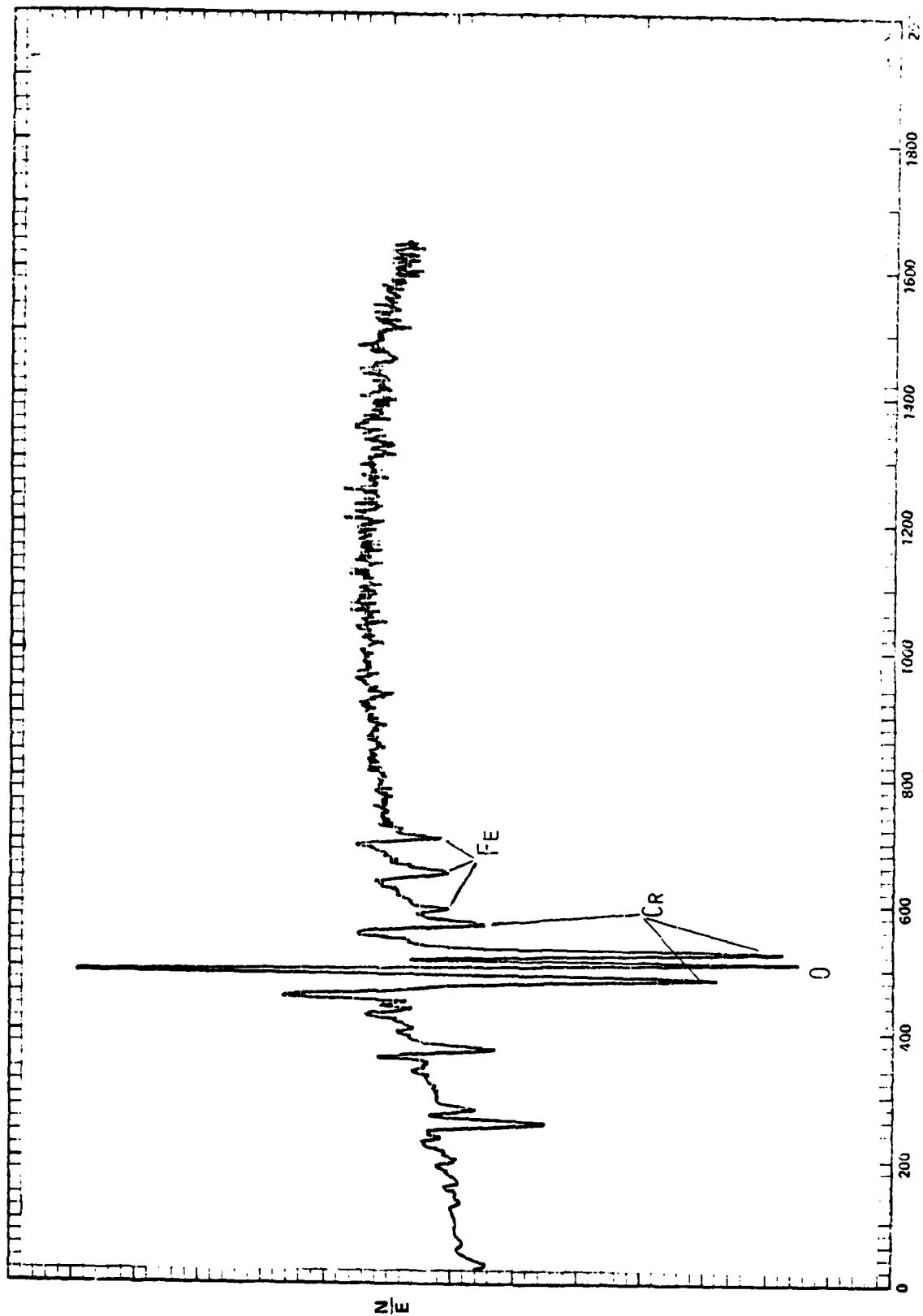


Figure 5. Auger Spectrum of the Surface of Specimen A-121.

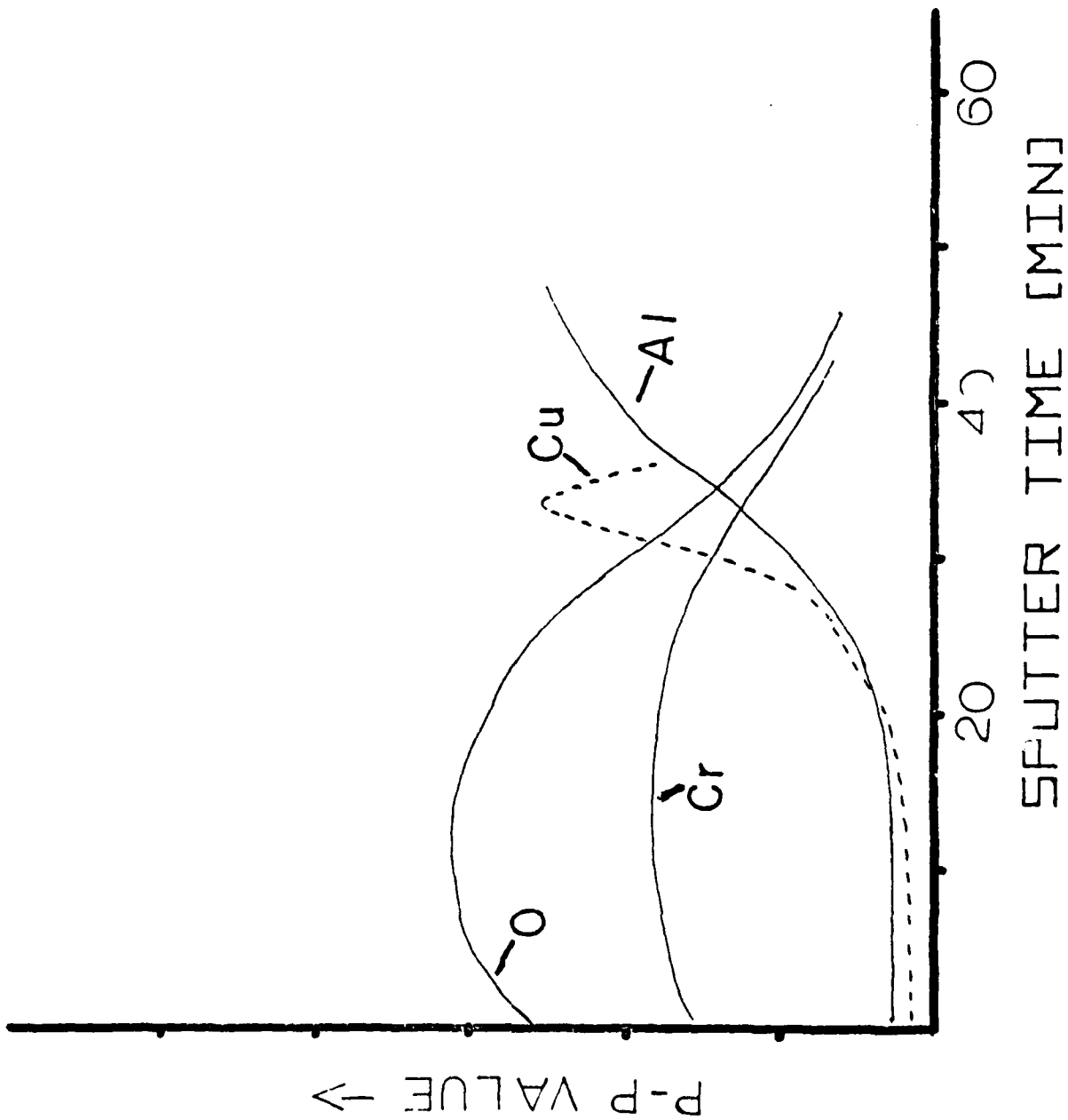


Figure 6. Auger Depth Profile of Specimen A-121.

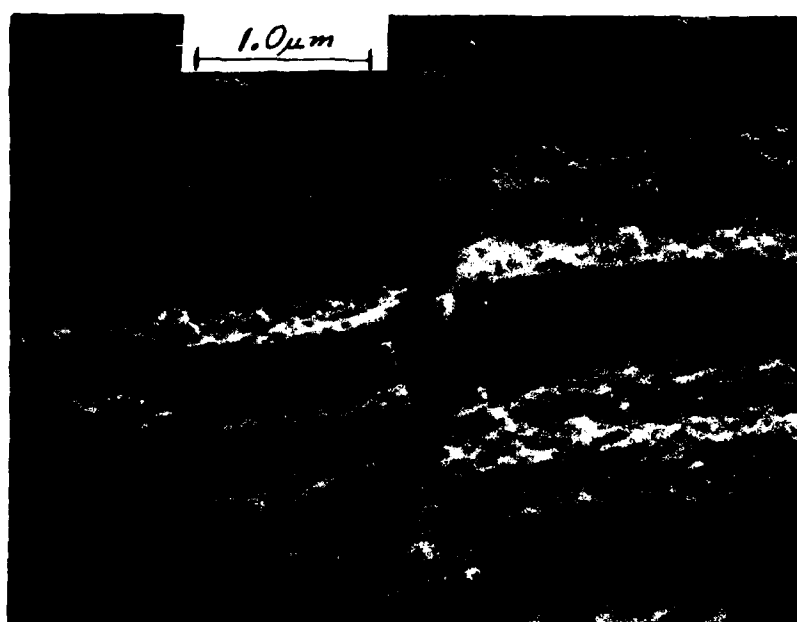


Figure 7. SEM Micrograph of Specimen AP-121.

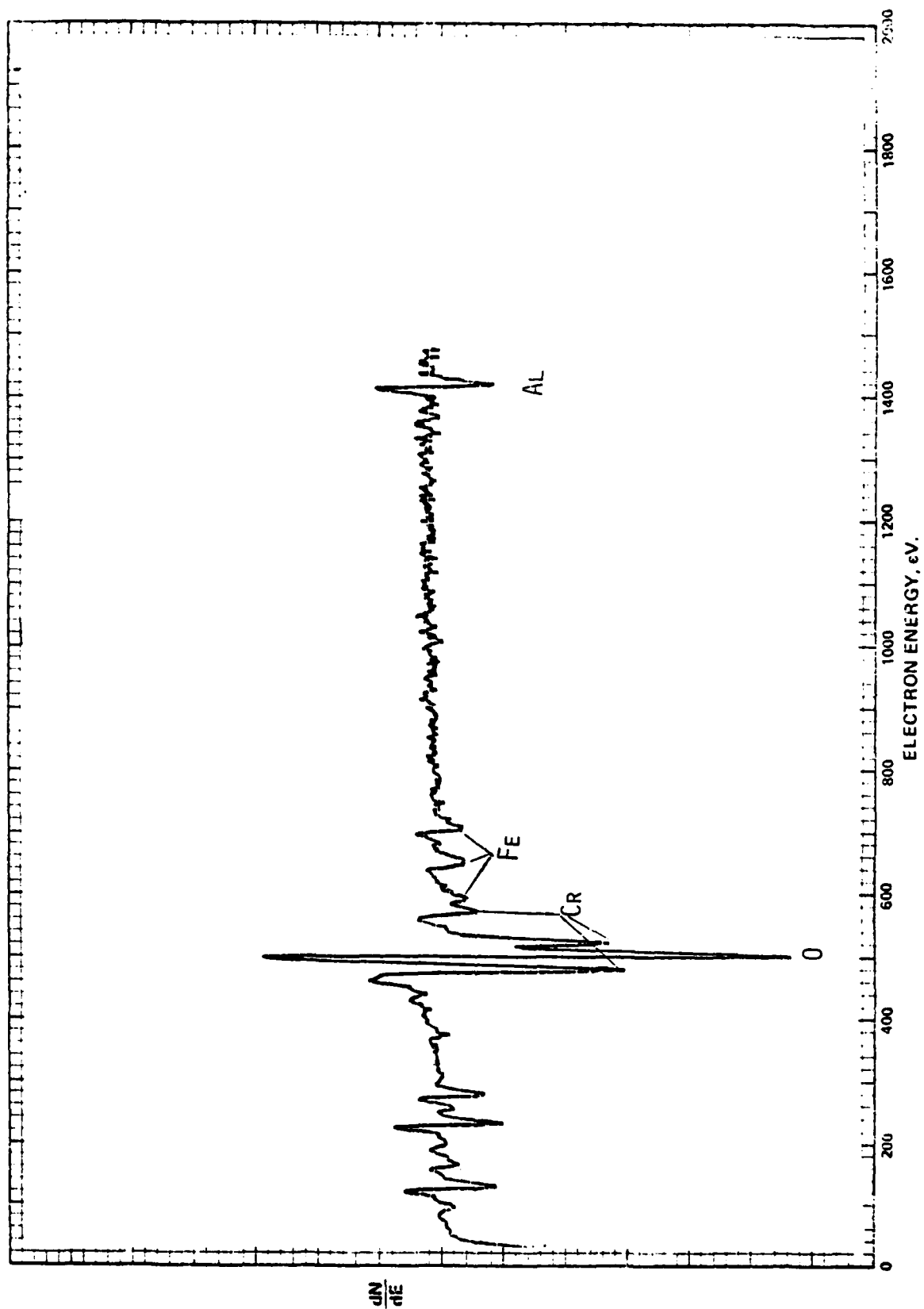


Figure 8. Auger Spectrum of the Surface of Specimen AP-121.

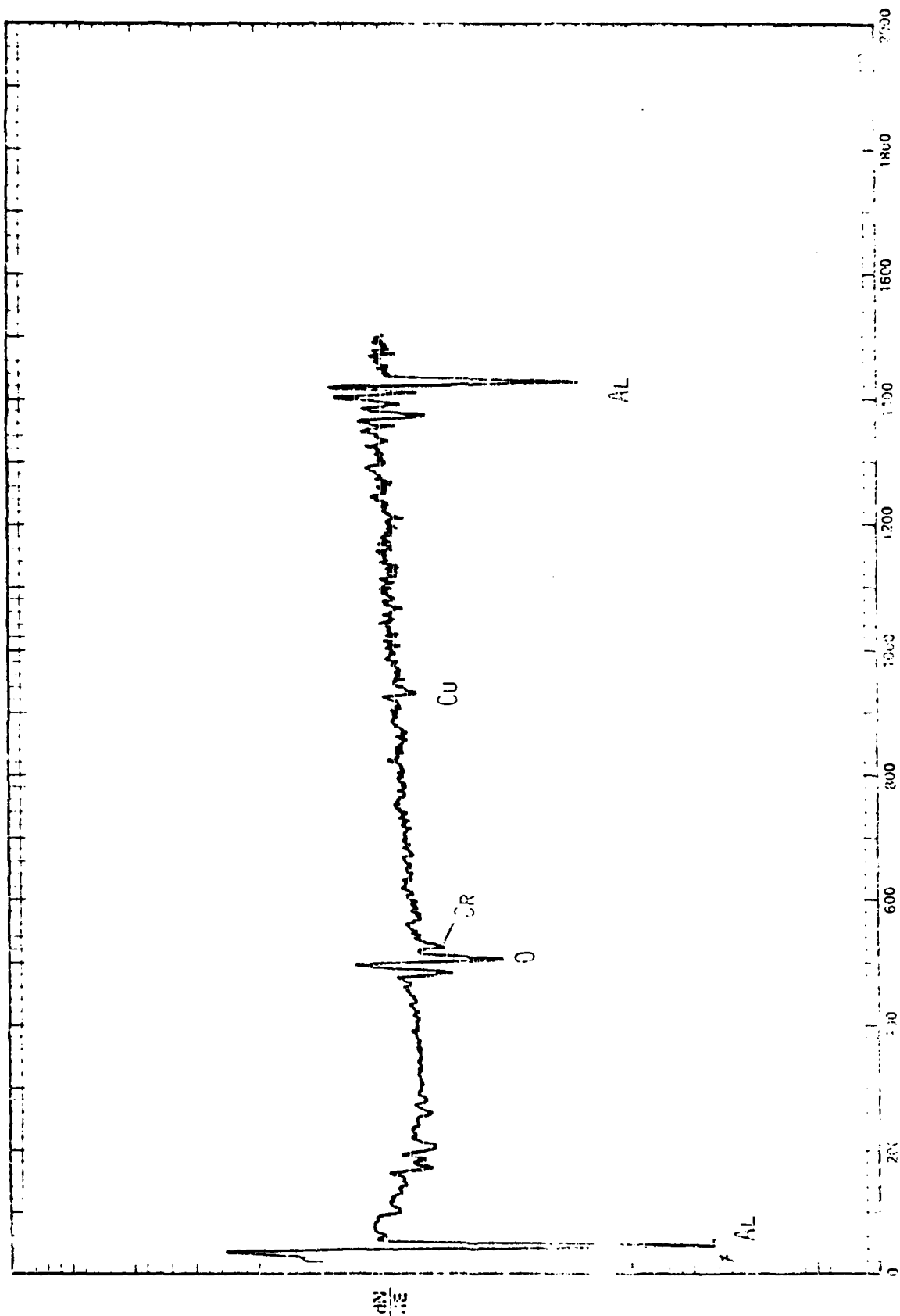


Figure 9. Auger Spectrum of Metal-Oxide Interface of Specimen AP-121.

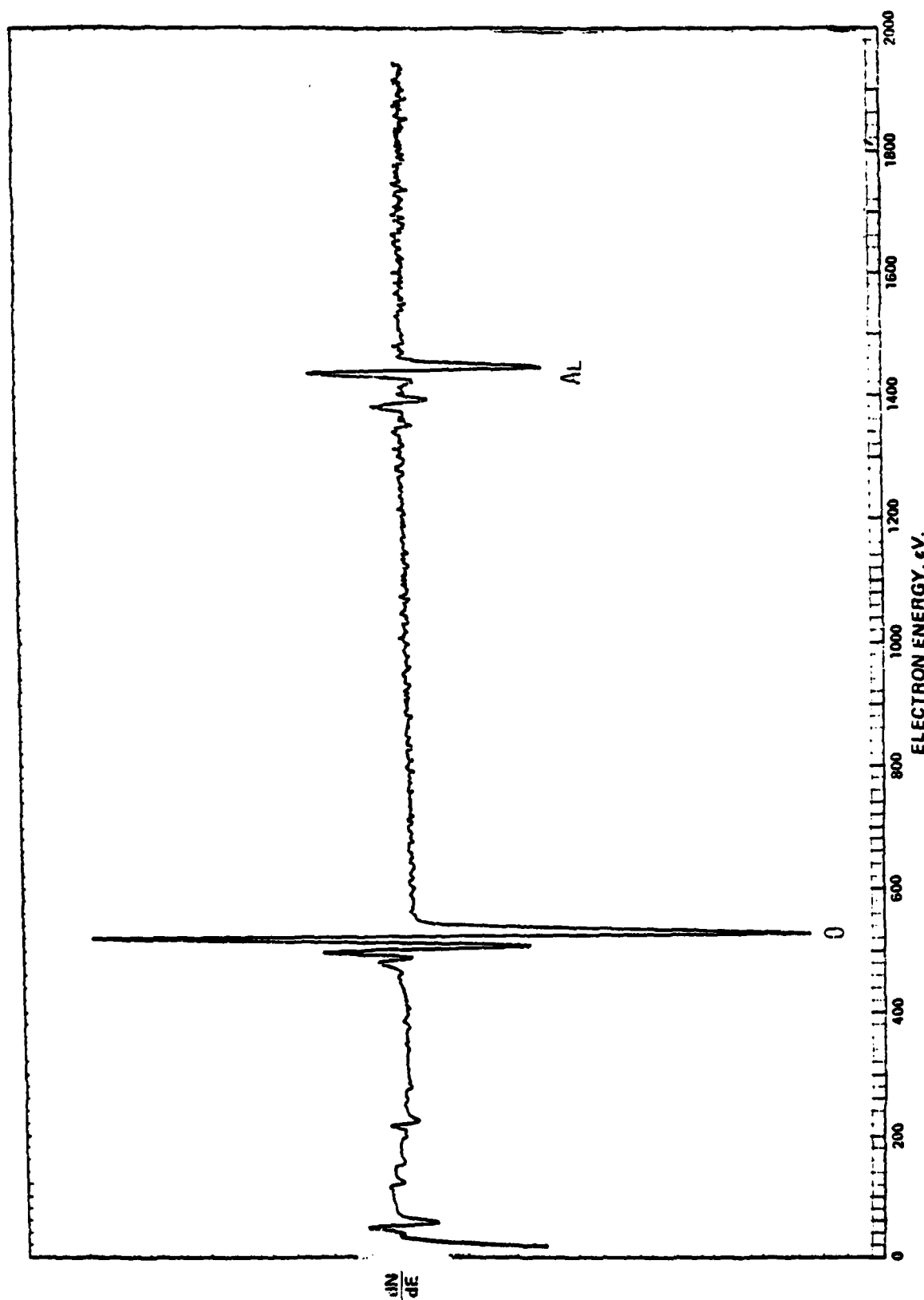


Figure 10. Auger Spectrum of the Surface of Specimen AP-095.

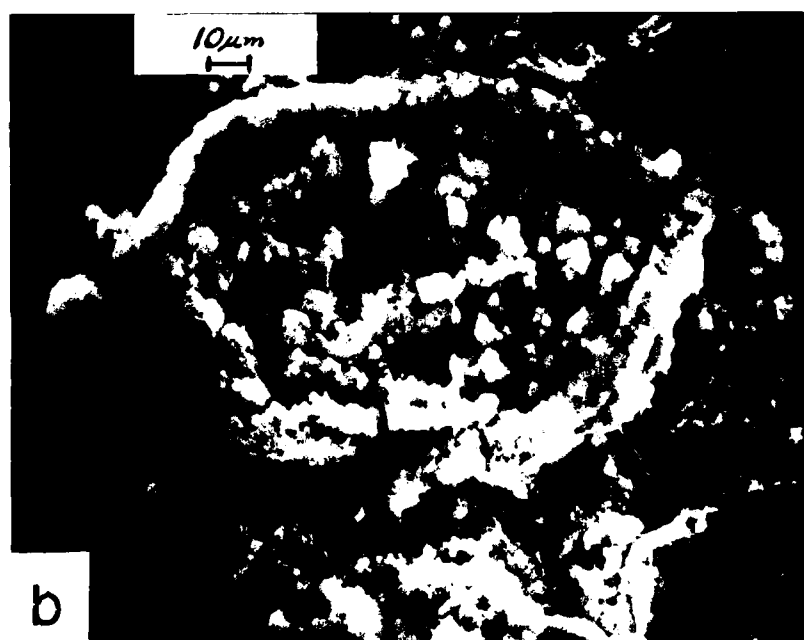


Figure 11. SEM Micrograph of Specimen P-036,
a) 100X, b) 500X.

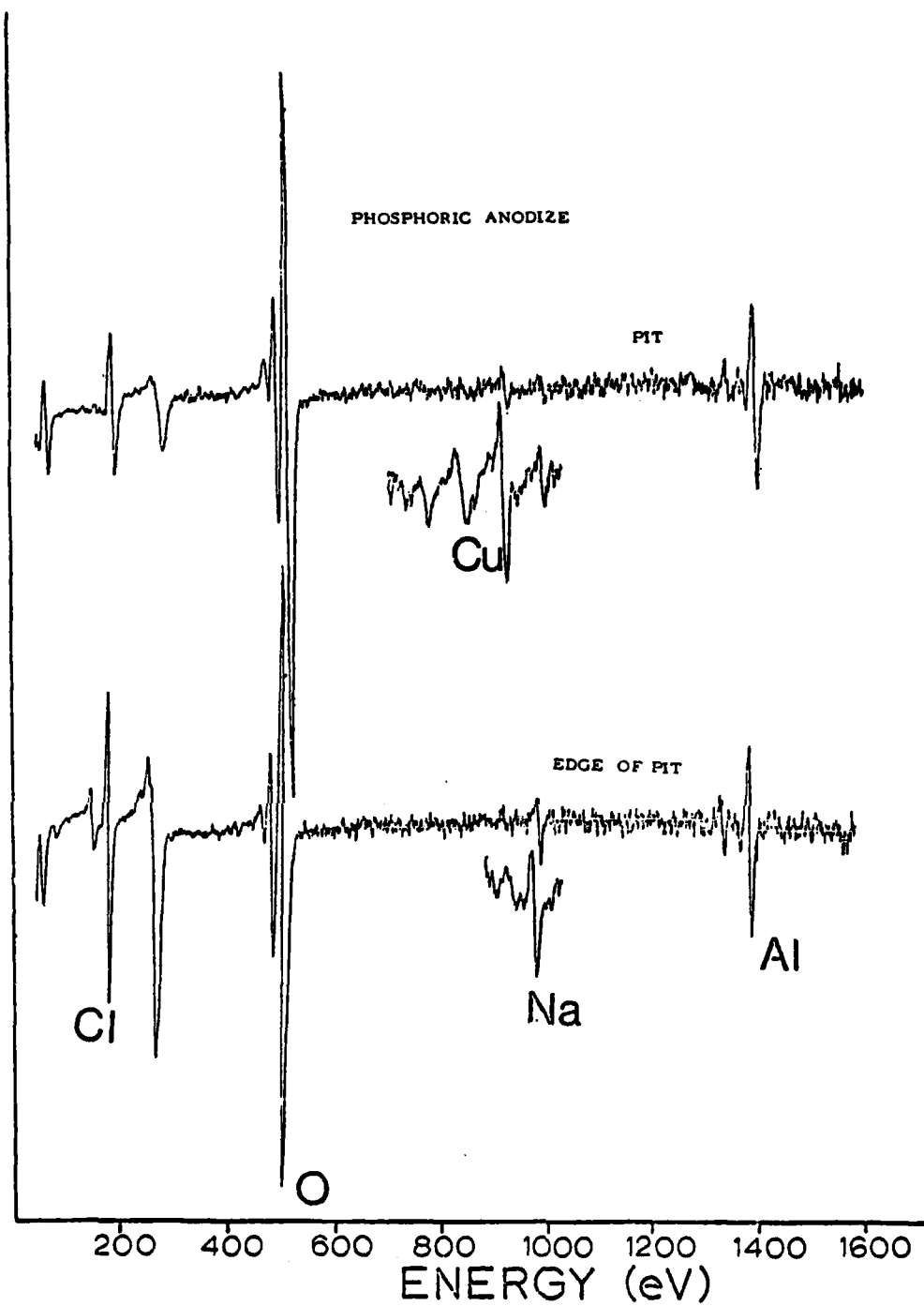


Figure 12. Auger Spectrum of Pit and Edge of Pit of Specimen P-036.

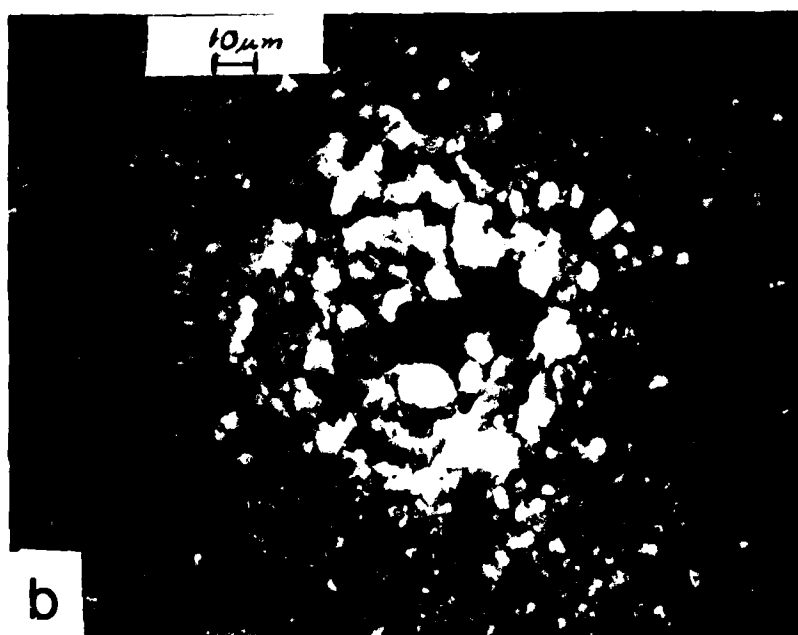


Figure 13. SEM Micrograph of Specimen A-036,
a) 100X, b) 500X.

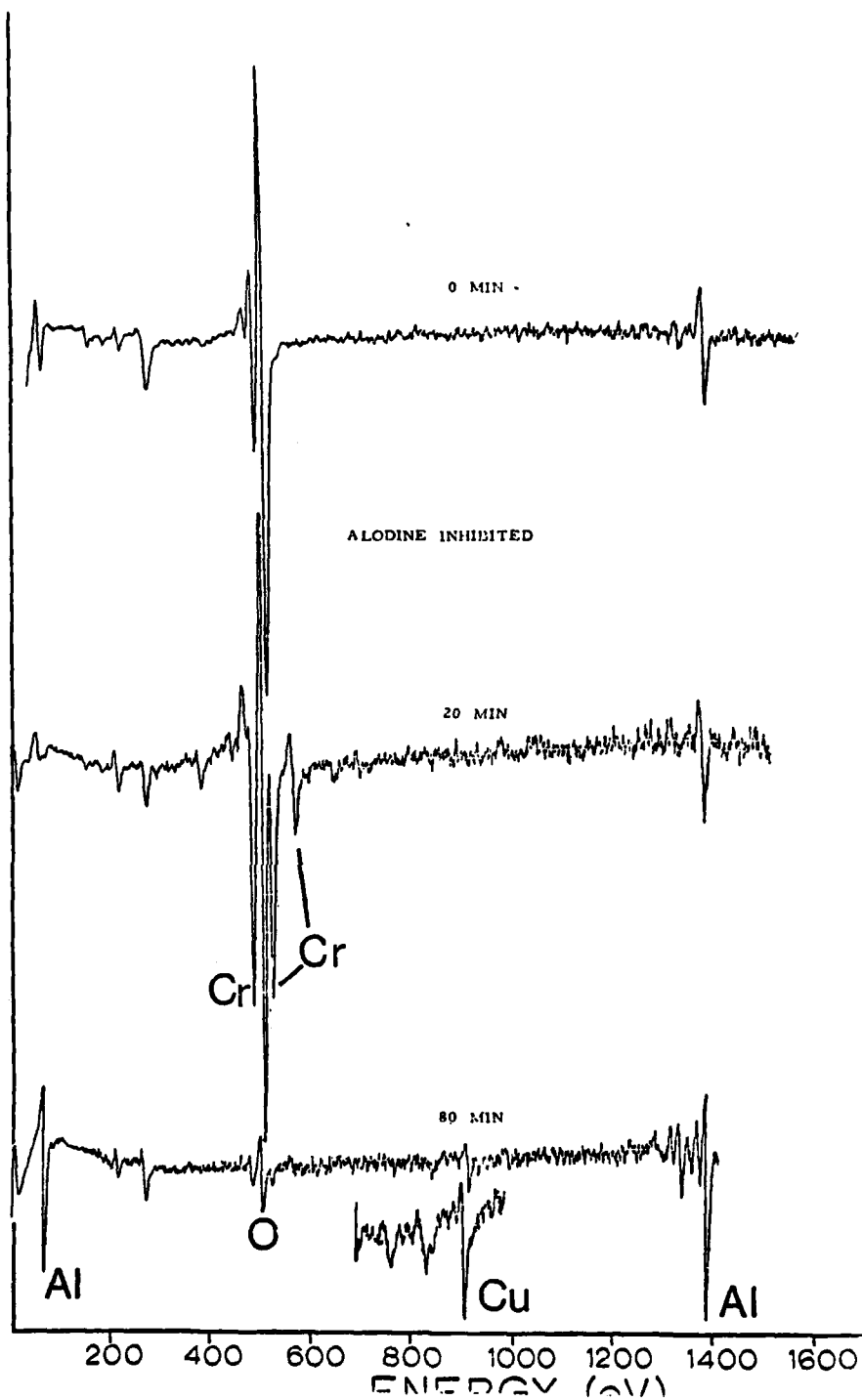


Figure 14. Auger Depth Profile Through Center of Healed Pit on Specimen A-036.

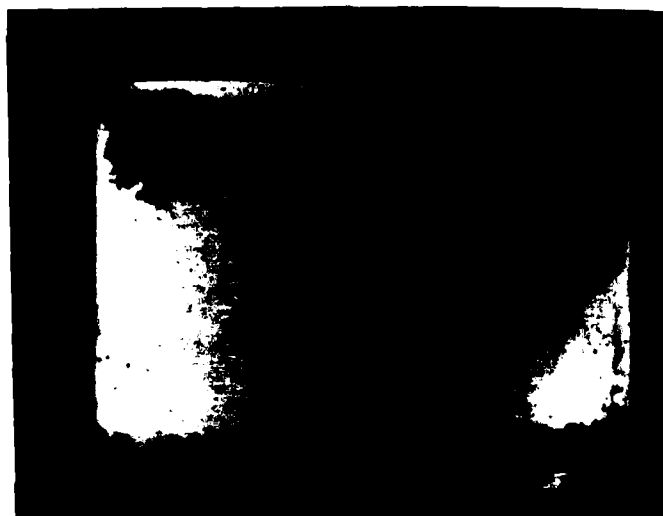


Figure 15. 3X Photo of Specimen AP-040 Showing
the Leaching Effect of Salt Solution.

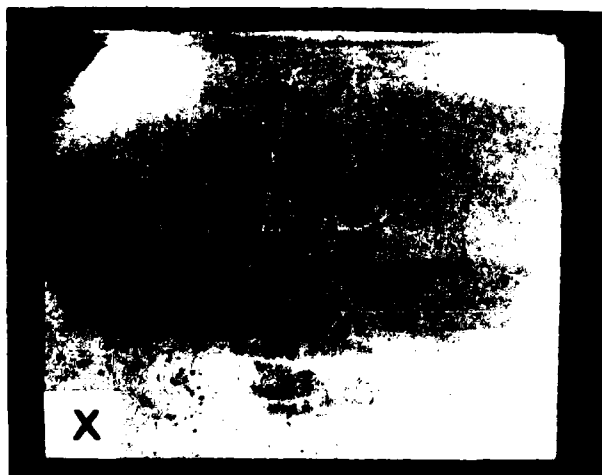


Figure 16. 3X Photo of Specimen AP-036, x) Sulfuric-Chromic Pretreatment, y) Nitric-Hydrofluoric Pretreatment.

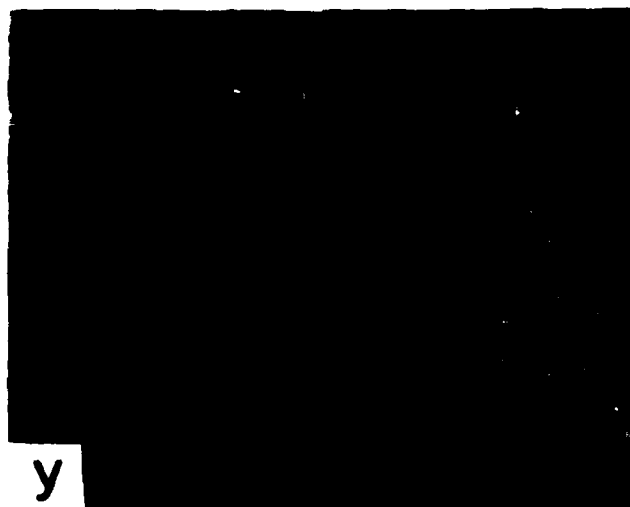


Figure 17. 3X Photo of Specimen AP-190, x) Sulfuric-Chromic Pretreatment, y) Nitric-Hydrofluoric Pretreatment.

TABLE I. EXPERIMENTAL CONDITIONS FOR SPECIMENS STUDIED.

SPECIMEN	PRETREATMENT	ALODINE	ANODIZATION	ENVIRONMENT
A-036	Nitric-HF	2 min		4% NaCl
P-036	Nitric-HF		7.5V, 5 min	4% NaCl
AP-036-5	Sulfuric-Chromic	4 min	7.5V, 5 min	4% NaCl
AP-036-8	Nitric-HF	4 min	7.5V, 5 min	4% NaCl
AP-040	Sulfuric-Chromic	4 min	10V, 10 min	1% NaCl
A-044	Sulfuric-Chromic	4 min		
AP-095	Nitric-HF	1 min	10V, 16 min	
A-121	Nitric-HF	1 min		
AP-121	Nitric-HF	1 min	40V, 4 min	
AP-190-10	Nitric-HF	2 min	10V, 8 min	5% NaCl
AP-190-22	Sulfuric-Chromic	2 min	10V, 8 min	5% NaCl

